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ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND WATER--ETC F/6 11/3  
IMPROVED ELECTRODEPOSITED LOW CONTRACTION CHROMIUM. (U)

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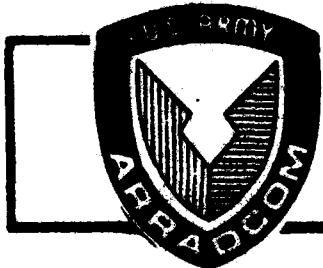
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TECHNICAL REPORT ARLCB-TR-82009

IMPROVED ELECTRODEPOSITED LOW CONTRACTION CHROMIUM

E. S. Chen

April 1982



US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND  
LARGE CALIBER WEAPON SYSTEMS LABORATORY  
BENET WEAPONS LABORATORY  
WATERVLIET, N. Y. 12189

AMCNS No. 611102H600011

DA Project No. 1L161102AH60

PRON No. 1A2250041AIA

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
ARLCB-TR-82009	AD-A118 678	
4. TITLE (and Subtitle)	5. TYPE OF REPORT & PERIOD COVERED	
IMPROVED ELECTRODEPOSITED LOW CONTRACTION CHROMIUM	Final	
7. AUTHOR(s)	6. PERFORMING ORG. REPORT NUMBER	
E. S. Chen		
9. PERFORMING ORGANIZATION NAME AND ADDRESS	10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS	
US Army Armament Research & Development Command Benet Weapons Laboratory, DRDAR-LCB-TL Watervliet, NY 12189	AMCMS No. 611102H600011 DA Project No. 1L161102AH60 PRON No. 1A2250041A1A	
11. CONTROLLING OFFICE NAME AND ADDRESS	12. REPORT DATE	
US Army Armament Research & Development Command Large Caliber Weapon Systems Laboratory Dover, NJ 07801	April 1982	
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office)	13. NUMBER OF PAGES	
	18	
16. DISTRIBUTION STATEMENT (of this Report)	15. SECURITY CLASS. (of this report)	
Approved for public release; distribution unlimited.	UNCLASSIFIED	
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, If different from Report)	15a. DECLASSIFICATION/DOWNGRADING SCHEDULE	
18. SUPPLEMENTARY NOTES		
Submitted for publication to Plating and Surface Finishing - American Electroplating Society.		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number)		
Chromium Plating	Microhardness	
Low Contraction Chromium	Electroactive Chemical	
Tensile Strength	Impurity Codeposition	
20. ABSTRACT (Continue on reverse side if necessary and identify by block number)		
A systematic study was made to optimize the parameters for plating high quality LC chromium using standard chromic acid solutions and solutions containing vanadium additions. The results indicate that deposit strengths were improved substantially by aging the plating solution at 250 A hr/liter and by using current densities in excess of 120 A/dm <sup>2</sup> . Under these conditions, hydrogen incorporation was reduced five-fold while hardness, strength, and cathode		
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20. ABSTRACT (CONT'D)

efficiency were maximized. A further improvement in deposit strength was obtained by adding vanadium as V<sub>2</sub>O<sub>5</sub> at a concentration of 10 g/l to standard chromic acid solutions. At higher concentrations, inferior deposits were produced that showed surface roughness, porosity, low strength, and higher hydrogen content.

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#### ACKNOWLEDGEMENTS

The author wishes to thank Mr. Richard Carter for preparing the chromium electrodeposits and performing the necessary mechanical tests and to acknowledge Mrs. Theresa Brassard for the metallographic work and hardness measurements.

## INTRODUCTION

The use of electrodeposited chromium coatings to protect metals is a well established industrial practice. For example, coatings of bright or hard chromium are routinely applied on various products to reduce galling, friction, wear, and erosion. For corrosion protection, a softer, crack-free variety of electrodeposited chromium has been used with good results.<sup>1</sup> This type of chromium, designated "LC" for low contraction, may be readily plated from a standard chromic acid bath.

Thick coatings of LC chromium have been used in ordnance<sup>2,3</sup> and other applications<sup>4,5</sup> such as gun tubes and engine cylinders where extreme conditions of corrosion and wear are encountered. The results, while encouraging, were inconsistent and improvements may be effected by optimizing the quality of the deposits.

In the present study, experiments were carried out to upgrade the LC chromium plating process. Major emphasis was directed towards improving deposit strength through the use of optimum plating parameters and adding electroactive chemicals to the plating solutions. The selection of V<sub>2</sub>O<sub>5</sub> for this purpose was based on an earlier report where V<sub>2</sub>O<sub>5</sub> showed a beneficial influence on the electrodeposition of cobalt.<sup>6</sup> Subsequent voltammetric

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<sup>1</sup>W. H. Safranek, The Properties of Electrodeposited Metals and Alloys, American Elsevier Publishing Co., Inc., (1974), p. 33.

<sup>2</sup>J. P. Young, NBS Report No. 3B10i, August 1, 1954.

<sup>3</sup>V. A. Lamb and J. P. Young, NBS Technical Note 46, May 1960.

<sup>4</sup>W. H. Safranek, The Role of Coatings in the Prevention of Mechanical Failures, (1976), pp. 56-63.

<sup>5</sup>The Iron Age, Y. 175, No. 2D, (1955), p. 140.

<sup>6</sup>G. R. Lakshminarayanan, E. S. Chen, J. C. Sadak, and F. K. Sautter, J. Electrochem. Soc., Vol. 123, No. 11 (1976), p. 1612.

measurements revealed its potential to diminish the reduction of hexavalent chromium and the formation of hydrogen in chromic acid solutions.

#### EXPERIMENTAL PROCEDURES

##### Plating Procedure

All chemicals were of reagent grade and used without further purification. A chromium stock solution was prepared and stored in two portions by dissolving 250 g/l of chromic acid and 2.5 g/l of sulfuric acid in double distilled water. The first was used without additional refinements while the second was pre-electrolyzed (aged) 250 A hr/l prior to use. V<sub>2</sub>O<sub>5</sub> at concentrations of 10, 15, and 20 g/l was added to the aged solutions.

Because of the brittleness of electrodeposited chromium, tensile test specimens were electroformed in the shape of cylinders. The electroforms were plated at a solution temperature of 85 ± 0.5°C using current densities ranging from 60 to 150 A/dm<sup>2</sup>. The plating was conducted in one liter Pyrex beakers using Teflon-coated magnetic stirrers for agitation. Rotating brass tubes having a masked area of 10 cm<sup>2</sup> were used as cathodes. A platinum anode with a surface area of 20 cm<sup>2</sup> was formed in a convex shape with respect to the cathode to provide constant current density throughout the cathode surface. Electroformed chromium tubes were obtained for evaluation by dissolving the brass in dilute nitric acid solutions.

##### Physical and Mechanical Property Measurements

Tensile strengths were measured with a table model Instron tensile machine. To minimize damage through handling and during testing, special fixtures were devised to grip the brittle chromium tubes. PVC end caps were

machined to accommodate the tubular samples and quick setting epoxy was used to cement them in position. The total lengths of the assembled test sample and the gage section were 7.5 and 3.5 cm respectively. The test sample was mounted on the universal coupling of the Instron tester by passing a pin through the sample end cap and the coupling. Stress was applied to the specimen by lowering the movable Instron grip until its opened jaws engaged the shoulders of the lower end cap, Figure 1. A crosshead speed of 0.05 cm/min was used in all the tests.

The hardness was measured with a Knoop diamond indenter at a load of 50 g.

Codeposited hydrogen and oxygen were determined using vacuum fusion and nitrogen by semi-micro Kjeldahl analysis.

#### RESULTS AND DISCUSSION

Figure 2 shows the plating rate of LC chromium as a function of current density for deposits plated from unaged solutions (2A), pre-electrolyzed solutions (2B), and solutions containing 10 g/l V<sub>2</sub>O<sub>5</sub> (2C). The plating rates were determined from the weights of the deposits and the use of an average LC chromium density value of 7.15 g/l. In all cases, the plating rate increased with increasing current density. The higher plating rates were obtained with pre-electrolyzed solutions. At a current density of 150 A/dm<sup>2</sup>, the deposition rate was as high as 3.5 times that associated with conventional bright chromium.

The results of the cathode efficiency measurements as a function of current density for the three different chromium plating solutions are given in

Figure 3. The cathode efficiency is obtained from the weight of the chromium deposited on the basis that hexavalent chromium ion is reduced to metallic chromium. The cathode efficiency obtained with pre-electrolyzed solutions, Figure 3B, varies between 11.1 and 13.1 percent and is 2 to 3 percent higher than that obtained with the unaged solutions, Figure 3A. In both cases, the efficiency increases with current density from  $60 \text{ A/dm}^2$  and reaches a plateau at a current density of  $120 \text{ A/dm}^2$ . With solutions containing  $10 \text{ g/l V}_2\text{O}_5$ , the efficiency also increased with current density. However, unlike the two previous solutions, a maximum efficiency of 12.5 percent is reached at a current density of  $90 \text{ A/dm}^2$ . Solutions containing 15 and  $20 \text{ g/l V}_2\text{O}_5$  showed maximum efficiencies of 8.0 and 5.0 percent respectively.

The hardness, tensile strength, and codeposited oxygen, hydrogen and nitrogen for LC chromium are given in Table I. At a given current density, the hardness decreases in the order of deposits from unaged solutions > deposits from pre-electrolyzed solutions > deposits from solutions containing  $10 \text{ g/l V}_2\text{O}_5$ . In all three cases, the hardness ranged from 525 to 668 KHN for current densities between 60 and  $150 \text{ A/dm}^2$ . Chromium deposits plated from solutions containing 15 and  $20 \text{ g/l V}_2\text{O}_5$  are softer by as much as 174 KHN.

LC chromium plated from unaged solutions exhibits very low tensile strengths. From Table I and Figure 4, the tensile strength is seen to increase from 7.1 to 29.1 ksi with increasing current density from 60 to  $150 \text{ A/dm}^2$ . Pre-electrolyzing the plating solution raises the tensile strength of all deposits appreciably. For the same current density range, the tensile strength increases from 32.8 ksi to a maximum of 48.2 ksi (at  $135 \text{ A/dm}^2$ ) followed by a decrease to 44.0 ksi. The addition of  $\text{V}_2\text{O}_5$  to the plating solution

at a concentration of 10 g/l improves the tensile strength to between 54.1 and 56.3 ksi. The use of higher concentrations of V<sub>2</sub>O<sub>5</sub>, 15 and 20 g/l, had the negative effect of lowering the tensile strength.

The codeposition of oxygen, hydrogen, and nitrogen is very much affected by the various plating parameters. With increasing current density, deposits plated from pre-electrolyzed solutions demonstrate that nitrogen content decreases from 0.006 to 0.004 wt.% while oxygen and hydrogen increase from 0.014 to 0.050 and from 0.0115 to 0.0137 wt.% respectively. In comparison, the magnitude of gas codeposition is considerably higher in coatings plated from unaged solutions; hydrogen and nitrogen levels as high as 0.0513 and 0.009 wt.% were observed. Deposits plated from solutions containing 10 g/l V<sub>2</sub>O<sub>5</sub> show the lowest content of gas incorporation for all three gases. However, at V<sub>2</sub>O<sub>5</sub> concentrations of 15 and 20 g/l, the trend reverses and large amounts of hydrogen and oxygen as occluded electrolytes are introduced into the deposits. The severity of the occlusions increases with current density and V<sub>2</sub>O<sub>5</sub> concentrations as illustrated in Figure 5. A consequence of occlusions is the reduction of hardness and tensile strength mentioned earlier.

The codeposition of gases can have both beneficial and detrimental effects on the properties of LC chromium deposits. For example, the presence of oxygen in the form of basic inclusions can increase deposit hardness through the mechanism of dispersion-strengthening in a manner similar to that observed in the precipitation hardening of metals. The increase in hardness with oxygen content reported in Table I can be explained in this fashion.

TABLE I. HARDNESS, TENSILE STRENGTH AND CODEPOSITED OXYGEN, HYDROGEN, AND NITROGEN CONTENT  
IN LG CHROMIUM ELECTRODEPOSITED AT 85°C.

Standard Chromium Bath	Current Density (A/dm <sup>2</sup> )	Oxygen (wt.%)	Hydrogen (wt.%)	Nitrogen (wt.%)	Weight Ratio O <sub>2</sub> /H <sub>2</sub>	Free <sup>c</sup> Hydrogen (wt.%)	Hardness (KHN)	Tensile Strength (ksi)
Unaged <sup>a</sup>	60	0.036	0.0513	0.007	0.7	0.0468	562	7.1
	90	0.021	0.0244	0.009	0.9	0.0218	620	13.6
	120	—	—	—	—	—	655	20.1
	150	0.031	0.0304	0.006	1.0	0.0265	668	29.1
Pre-electrolyzed <sup>b</sup>	60	0.014	0.0115	0.006	1.2	0.0097	540	32.8
	90	0.033	0.0105	0.006	3.1	0.0064	578	40.0
	120	0.034	0.0096	0.005	3.4	0.0050	646	47.0
	135	0.048	0.0132	0.004	3.6	0.0072	643	48.2
	150	0.050	0.0138	0.004	3.6	0.0075	657	45.5
With V <sub>2</sub> O <sub>5</sub> at 10 g/l	60	0.020	0.0042	0.002	4.8	0.0017	525	54.1
	90	0.028	0.0067	0.001	4.2	0.0032	562	46.7
	120	0.038	0.0108	0.001	3.5	0.0060	581	47.0
	135	0.031	0.0102	0.001	3.0	0.0063	617	44.8
	150	0.040	0.0054	0.001	7.4	0.0004	640	56.3
With V <sub>2</sub> O <sub>5</sub> at 15 g/l	60	0.075	0.0090	0.001	8.3	0.0000	495	9.5
	90	0.062	0.0112	0.001	5.5	0.0034	549	38.1
	120	0.101	0.0148	0.001	6.8	0.0022	586	44.2
	135	0.087	0.0112	0.001	7.8	0.0003	596	34.4
	150	0.096	0.0159	0.001	6.0	0.0039	609	30.4
With V <sub>2</sub> O <sub>5</sub> at 20 g/l	60	0.062	0.0080	0.001	7.8	—	535	15.5
	90	0.137	0.0101	0.001	13.6	—	526	33.1
	120	0.182	0.0120	0.001	15.2	—	520	43.4
	135	0.311	0.0231	0.001	14.8	—	480	23.4
	150	0.365	0.0234	0.002	15.6	—	466	21.5

<sup>a</sup>The values of oxygen, hydrogen, and nitrogen content in these deposits are the result of a single measurement.

<sup>b</sup>Each value of oxygen content represents an average of three determinations. The values of hydrogen and nitrogen represent an average of two determinations.

<sup>c</sup>The free hydrogen was calculated on the basis of oxygen combining with hydrogen in a weight ratio of eight.

The process of electrodeposition can introduce large quantities of nitrogen in chromium deposits apart from that dictated by solubility data. It was reported that the nitrate ion concentration in a conventional chromic acid plating solution was reduced from 2.7 to 0.008 g/l after an electrolysis of 25 A hr/l. The corresponding nitrogen content in the chromium was reduced from 0.24 to 0.025 wt.%.<sup>7</sup> The presence of nitrogen is known to increase the ductile-brittle transition temperature. It was reported that by water-quenching chromium samples containing 80 ppm nitrogen the transition temperature was increased upwards to 250°C.<sup>8</sup>

Although the equilibrium solubility of hydrogen in chromium is extremely small, electrodeposited chromium can retain a large quantity of hydrogen. The mode of occurrence has been the subject of considerable speculation and the consensus is that hydrogen most probably exists in the form of hydrides and hydrated chromic oxides.<sup>6</sup> While both forms contribute to the buildup of stress, the unstable hydride formation and the shrinkage effect accompanying the hydride decomposition are believed to play a major role. Figure 6 shows an LC chromium deposit where the stress buildup is sufficient to cause spallation. This spallation is commonly obtained in deposits plated from unaged solutions at a current density of 60 A/dm<sup>2</sup>. Spallation does not occur in deposits plated at either a higher current density or from pre-electrolyzed solutions. It is of interest to note that the hydrogen content between spalled and coherent samples differs by factors of 2 to 5.

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6G. R. Lakshminarayanan, E. S. Chen, J. C. Sadak, and F. K. Sautter, *J. Electrochem. Soc.*, Vol. 123, No. 11 (1976), p. 1612.

7N. Ryan and E. J. Lumley, *J. Electrochem. Soc.*, Vol. 106, No. 5(1959), p. 388.

8A. H. Sully and E. A. Brandes, Chromium, New York Plenum Press (1967), p. 47, p. 170.

It was mentioned earlier that the increase in deposit hardness with increasing oxygen content is consistent with the view of dispersion-strengthening by codeposited basic inclusions. According to Brenner,<sup>9</sup> the basic inclusions most probably occur in the form of hydrated chromic acid,  $\text{Cr}_2\text{O}_3 \cdot x\text{H}_2\text{O}$ . Depending on the water of hydration, the weight ratio of oxygen to hydrogen can vary from 32 ( $\text{Cr}_2\text{O}_3 \cdot x\text{H}_2\text{O}$  where  $x = 1$ ) to a limiting ratio of eight for large values of  $x$ . Assuming the weight ratio of eight applies for combined hydrogen, the lower limit of all other "free" hydrogen in the deposits can be estimated. Table I shows that the estimated "free" hydrogen content is largest in deposits plated at low current densities and particularly from unaged solutions. A graph of "free" hydrogen versus tensile strength is given in Figure 7 showing the excellent correlation obtained with deposits from pre-electrolyzed solutions and from solutions containing 10 g/l V<sub>2</sub>O<sub>5</sub>. The graph indicates that the tensile strength of LC chromium can be optimized by reducing the concentration of "free" hydrogen in the deposits. In the present investigation, this was accomplished by solution aging and the addition of vanadium to the plating electrolyte. Other procedures such as the use of current modulation may also be effective. The relationship between tensile strength and "free" hydrogen is believed to be applicable to all chromium deposits which are free of plating defects; deposits with cracks, i.e. conventional hard chromium, spallations, voids or occluded electrolyte, do not fit in the correlation.

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<sup>9</sup>A. Brenner, P. Burhead, and C. Jennings, Res. Nat. Bur. Stand., 40 (1948), p. 31.

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3. V. A. Lamb and J. P. Young, NBS Technical Note 46, May 1960.
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6. G. R. Lakshminarayanan, E. S. Chen, J. C. Sadak, and F. K. Sautter, J. Electrochem. Soc., Vol. 123, No. 11 (1976), p. 1612.
7. N. Ryan and E. J. Lumley, J. Electrochem. Soc., Vol. 106, No. 5 (1959), p. 388.
8. A. H. Sully and E. A. Brandes, Chromium, New York Plenum Press (1967), p. 47, p. 170.
9. A. Brenner, P. Burhead, and C. Jennings, Res. Nat. Bur. Stand., 40 (1948), p. 31.

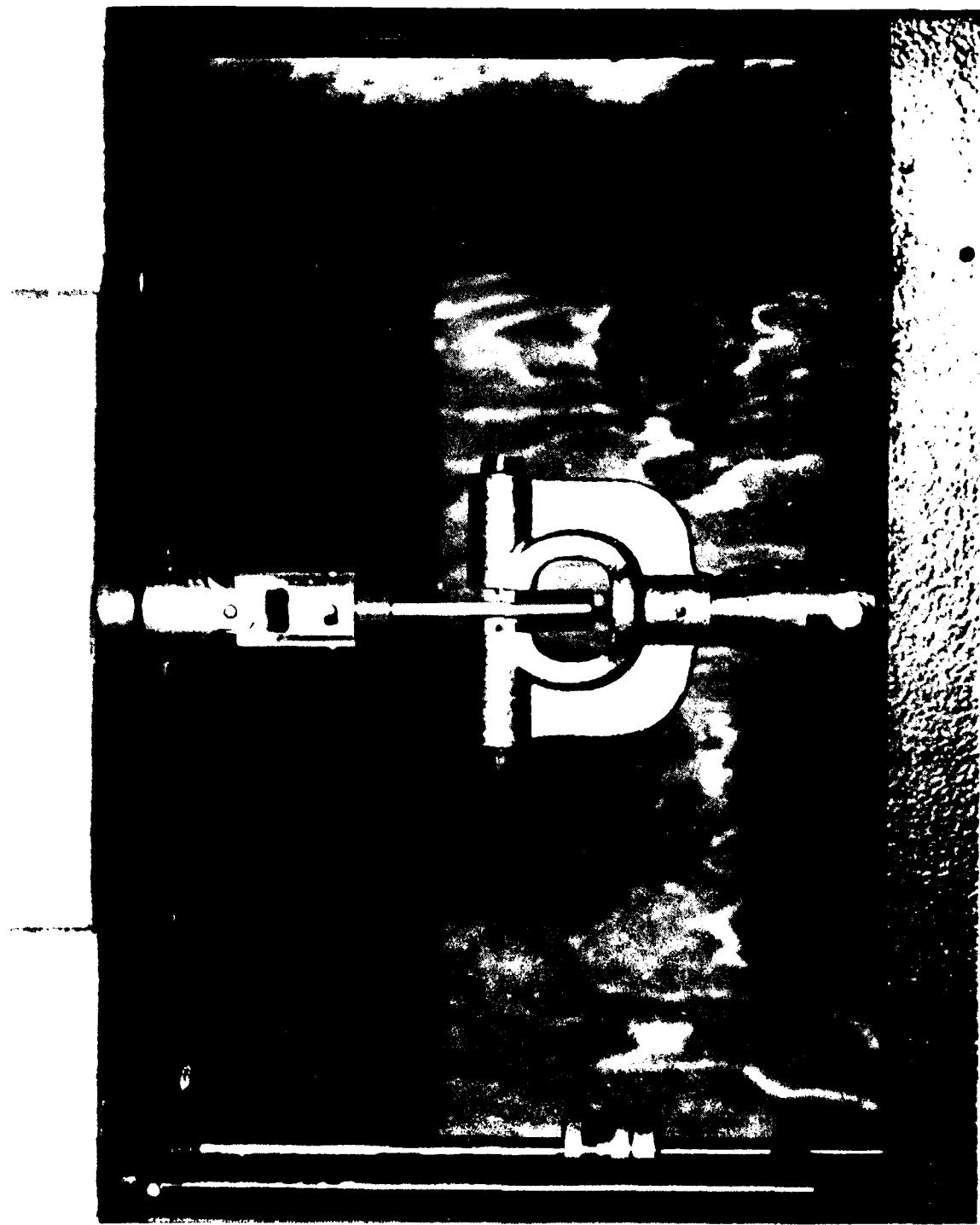


Figure 1. Tensile Strength Test.

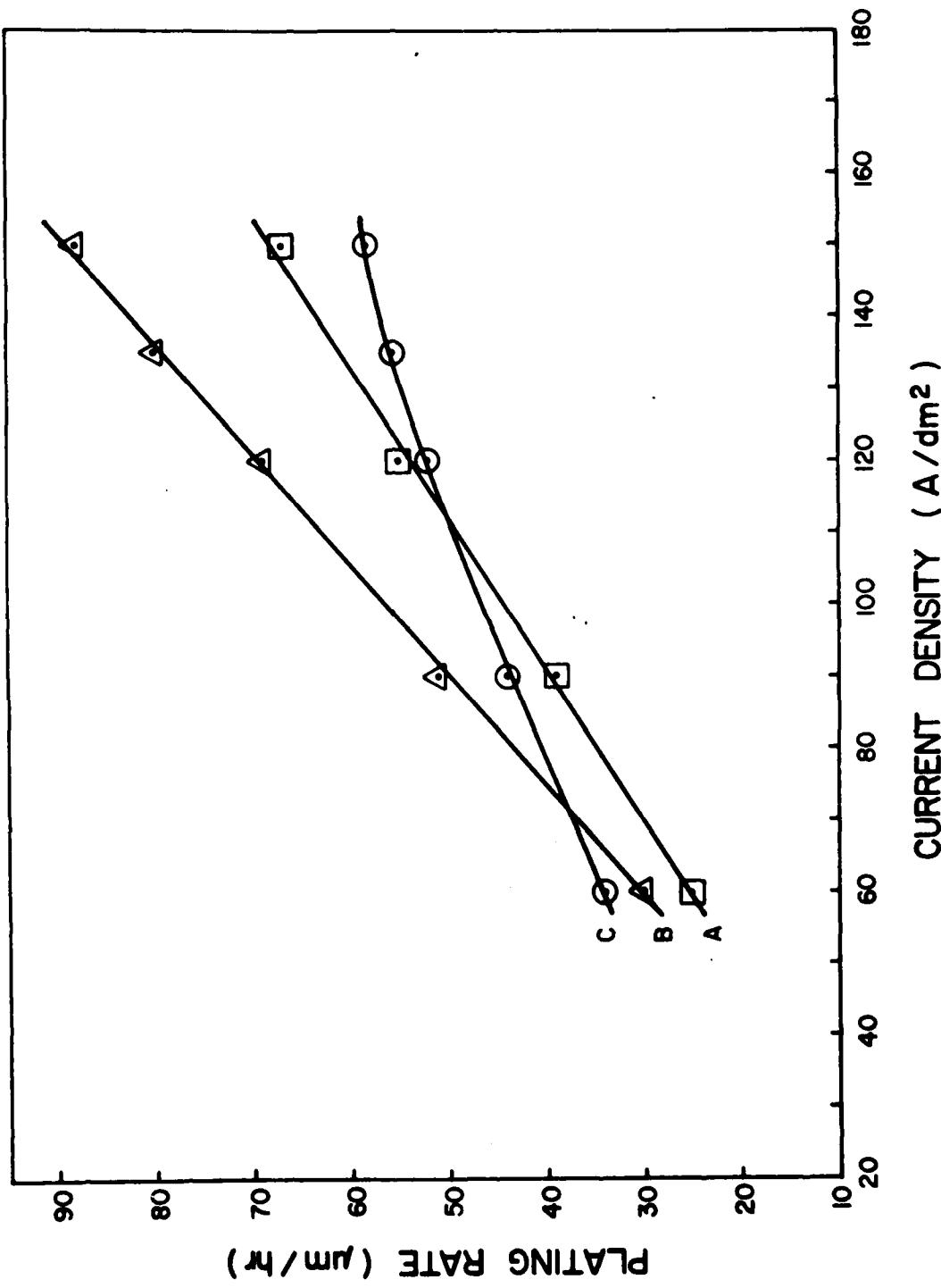


Figure 2. Plating Rate of LC Chromium with Current Density at 85°C.  
 A - unaged solution, B - pre-electrolyzed solution,  
 C - solution with 10 g/l  $\text{V}_2\text{O}_5$ .

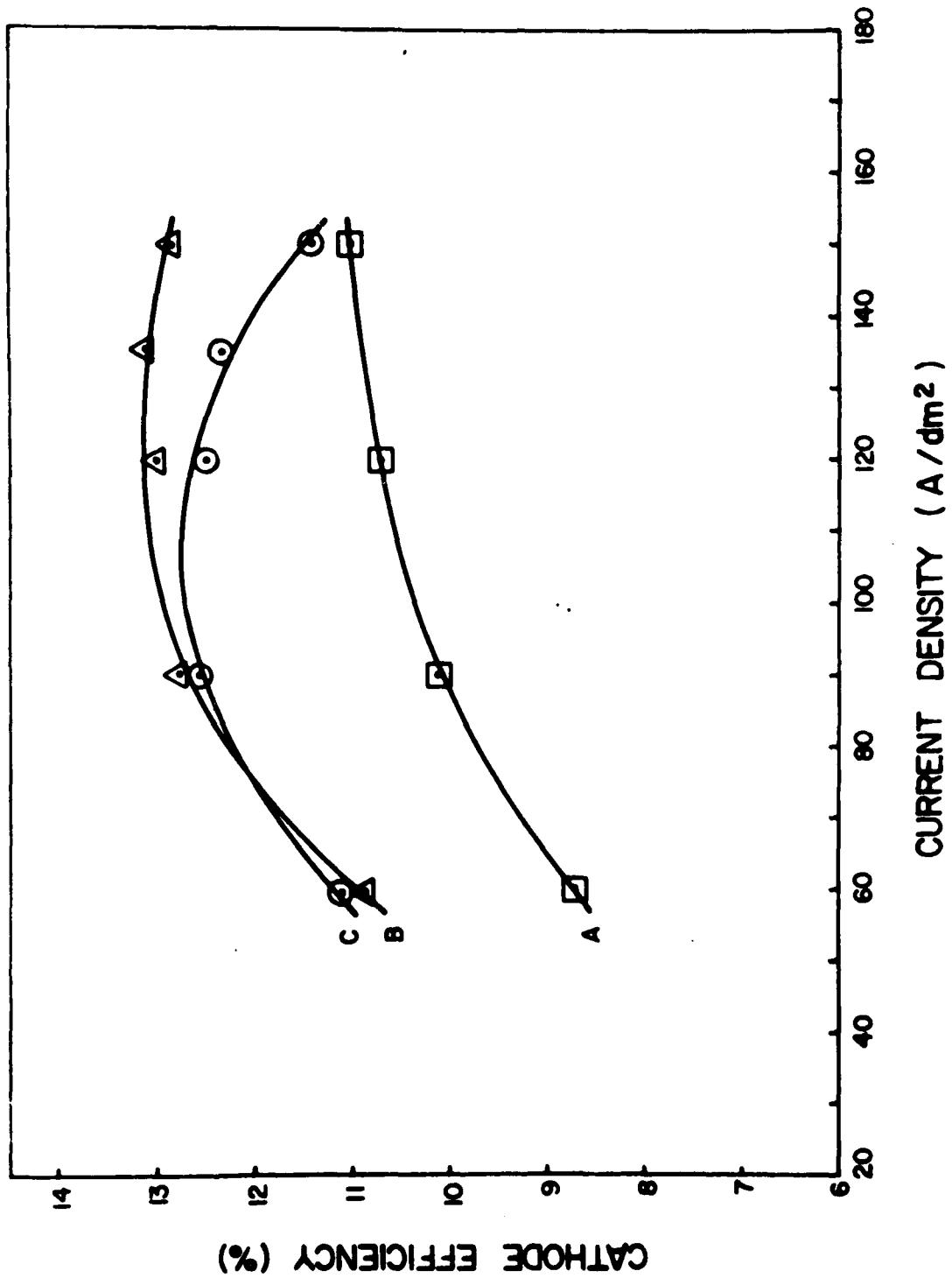


Figure 3. Cathode Efficiency of LC Chromium with Current Density at 85°C.  
 A - unaged solution, B - pre-electrolyzed solution,  
 C - solution with 10 g/l  $V_2O_5$ .

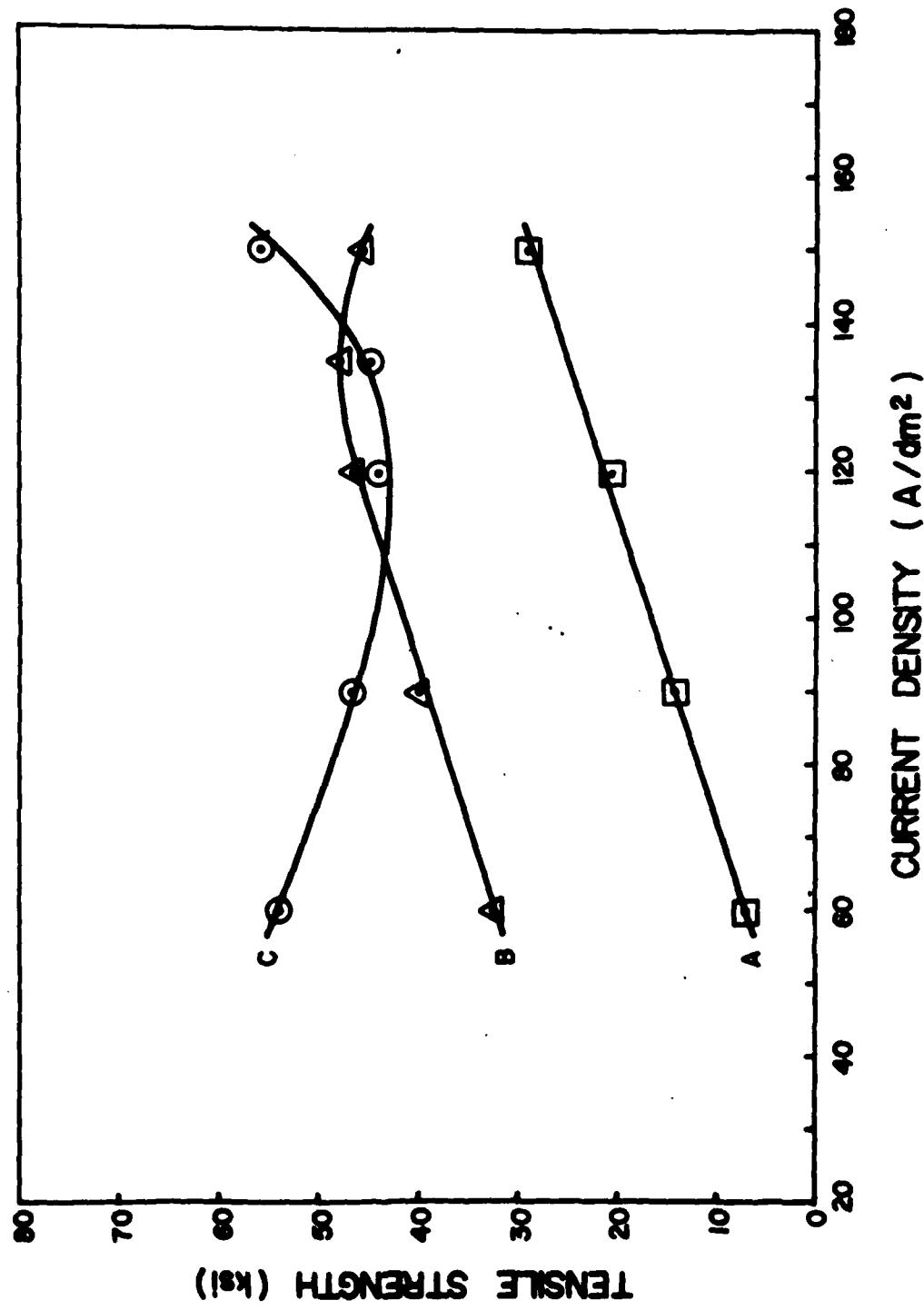
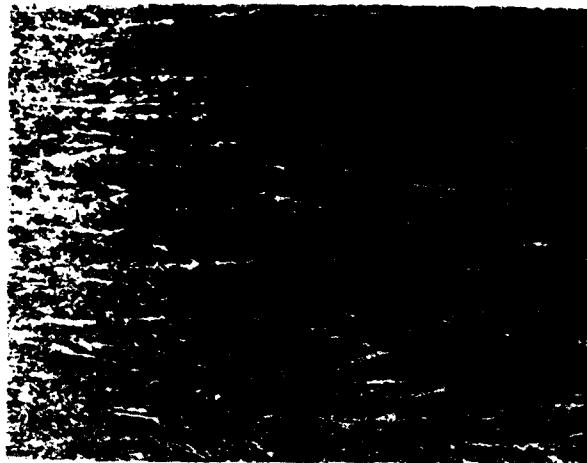


Figure 4. Tensile Strength of LC Chromium with Current Density at 85°C.  
 A - unaged solution, B - pre-electrolyzed solution,  
 C - solution with 10 g/l  $V_2O_5$ .



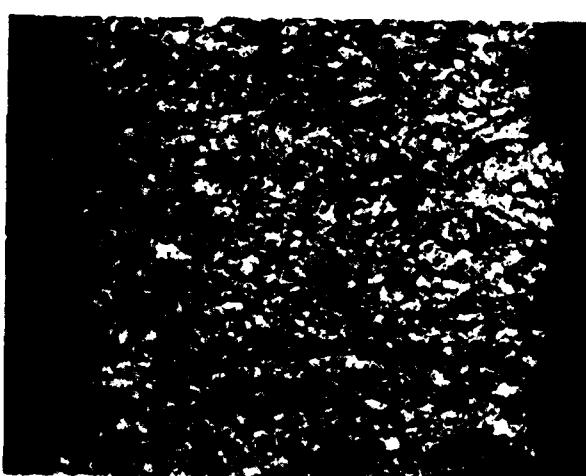
A



B



C



D

Figure 5. Microstructure of LC Chromium Deposited from Solutions Containing  $V_2O_5$  at 85°C. A - 10 g/l  $V_2O_5$ , 135 A/dm $^2$ ; B - 15 g/l  $V_2O_5$ , 120 A/dm $^2$ ; C - 20 g/l  $V_2O_5$ , 90 A/dm $^2$ ; D - 20 g/l  $V_2O_5$ , 135 A/dm $^2$ .

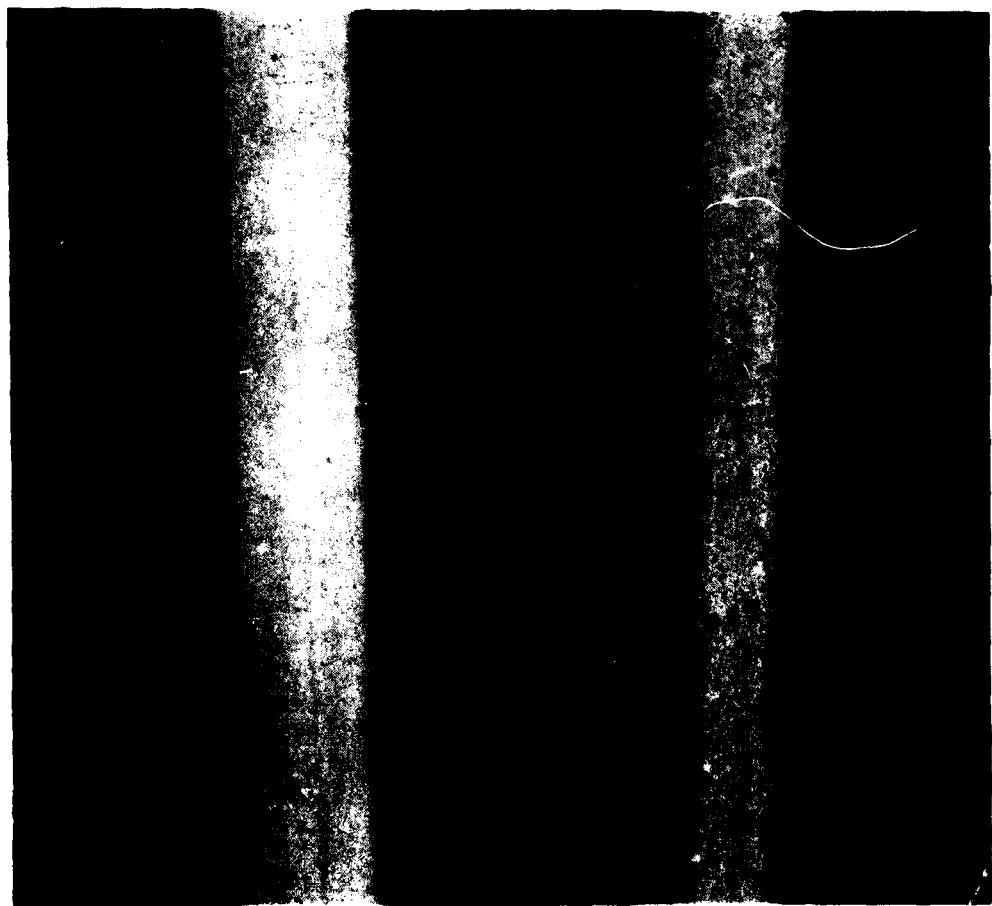


Figure 6. LC Chromium Plated from Unaged Solutions at 85°C. Left - good deposit, 120 A/dm<sup>2</sup>, Right - spalled deposit, 60 A/dm<sup>2</sup>.

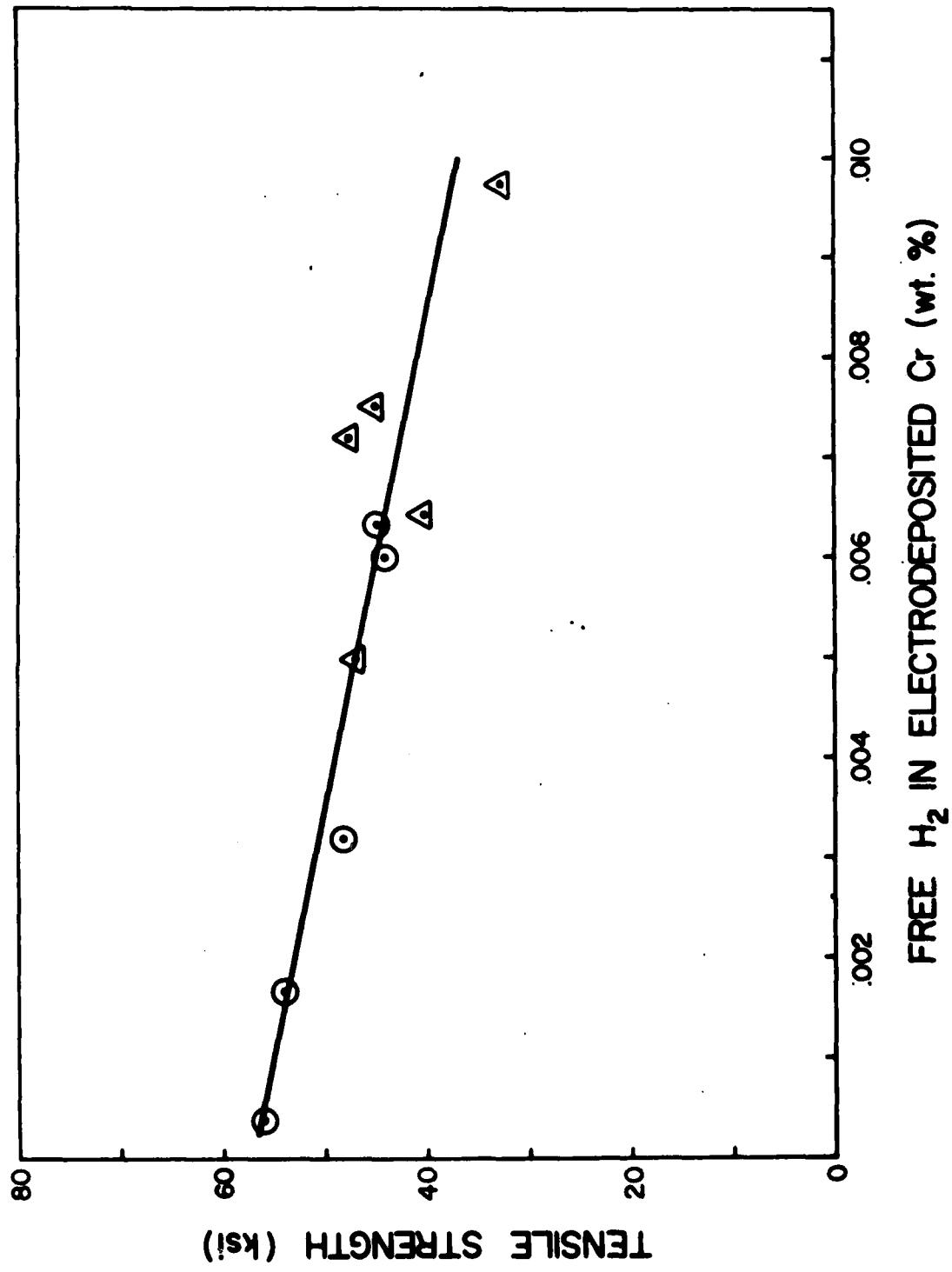


Figure 7. Tensile Strength of LC Chromium as a Function of Free Hydrogen in the Deposit. 0 - using solutions containing 10 g/l  $V_2O_5$ ,  $\Delta$  - using pre-electrolyzed solutions.

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